HiPIMS

A New Generation of Films Deposition Techniques for SRF Applications

A-M Valente-Feliciano

A. Anders (LBNL)
S. Calatroni, G. Terenzianini (CERN)
A. Ehiasarian (Sheffield Hallam Univ.)
L. Phillips (JLab)
Outline

- SRF films- State of the Art
- Energetic Condensation
- Energetic Condensation Techniques
- HiPIMS Principle
- HIPIIMS: an array of techniques
- Application to SRF Surfaces
- HiPIMS @ LBNL
- HiPIMS @ CERN
- HiPIMS @ JLAB
- Conclusions
Thin Films for SRF–state of the art

- CERN LEP 2  272 x 353MHz Nb/Cu 4-cell cavities
- INFN Legnaro  52 x 160 MHZ Nb/Cu QWR

1.5 GHz Nb/Cu cavities, sputtered w/ Kr @ 1.7 K \(Q_0=295/R_s\)

![Graph showing Q vs. accelerating field for Nb/Cu films](image)

- **Standard films**
  - RRR\(_{\text{max}}\) = 40
  - Columnar grains, size ~ 100 nm
  - In plane diffraction pattern: (110) fiber texture ⊥ substrate plane
- **Oxide-free films**
  - RRR\(_{\text{max}}\) = 28
  - Equi-axed grains, size ~ 1–5 \(\mu\)m
  - In plane diffraction pattern: zone axis \([110]\)

Heteroepitaxy
- Nb (110) // Cu(010)
- Nb (110) // Cu(111)
- Nb (100) // Cu(110)

Bulk Nb
- 1.5 GHz Nb/Cu
- 350MHz Nb/Cu (4.2K)

1.5 GHz Nb/Cu cavities, sputtered w/ Kr @ 1.7 K \(Q_0=295/R_s\)
Energetic Condensation

Condensing (film-forming) species: hyper-thermal & low energies (>10 eV).

Additional energy provided by fast particles arriving at a surface ⇒ number of surface & sub-surface processes ⇒ changes in the film growth process:

- residual gases desorbed from the substrate surface
- chemical bonds may be broken and defects created thus affecting nucleation processes & film adhesion
- enhanced mobility of surface atoms
- stopping of arriving ions under the surface

As a result of these fundamental changes, energetic condensation allows the possibility of controlling the following film properties:

- Density of the film
- Film composition
- Crystal orientation may be controlled to give the possibility of low-temperature epitaxy
Energetic Condensation Techniques

A variety of techniques with distinct technologies

- High Impulse Power Magnetron sputtering (HiPIMS)
- Vacuum Arc Plasma
- Coaxial Energetic Deposition (CED)
- Electron cyclotron Resonance (ECR)

WEIOA02 Thin Film Growth by Energetic Condensation
Mahadevan Krishnan - Alameda Applied Sciences Corporation
Energetic Condensation Techniques

A variety of techniques with distinct technologies

- High Impulse Power Magnetron sputtering (HiPIMS)
- Vacuum Arc Plasma
- Coaxial Energetic Deposition (CED)
- Electron cyclotron Resonance (ECR)

Engineering for optimum RF performance

3 sequential phases for film growth

- Film nucleation on the substrate
- Growth of an appropriate template for subsequent deposition of the final RF surface
- Deposition of the final surface optimized for minimum defect density.
HIPIMS: A Form of “Ionized Sputtering”
One Approach to Energetic Deposition

“What distinguishes HIPIMS from the long-practiced pulsed sputtering?”

Technical Definition:
HIPIMS is pulsed sputtering where the peak power exceeds the average power by typically two orders of magnitude.

(implies a long pause between pulses, hence the term “impulse”)

Physical Definition:
HIPIMS is pulsed sputtering where a very significant fraction of the sputtered atoms becomes ionized.

(implies that self-sputtering occurs, which may or may not be sustained by target ions)


Why do we care? Because bias can be applied to affect film-forming ions (not atoms)!

about 500 kW peak
15 cm dia. Cu target

image from the seminal (but not first) paper:
Probability for ion to return to target

Ionization probability

Probability for ion to return to target

Condition of self-sputtering runaway
\[ \Pi \equiv \alpha \beta \gamma > 1 \]

Condition of steady-state self-sputtering
\[ \Pi \equiv \alpha \beta \gamma = 1 \]
HiPIMS-High Power Impulse Magnetron Sputtering

High Impulse Power Magnetron Sputtering (HiPIMS) also known as HPPMS (High Power Pulse Magnetron sputtering)

Pulse with a **power density** at the target surface during the pulse exceeding the typical dc power density by about **two orders of magnitude**. This implies that the **off-time between pulses is long**, and the **duty cycle is only of order 1%**.

20 eV - 100 eV (vs. only about 2 -10 eV in conventional sputtering).

**Variation: Burst HiPIMS or Modulated Pulse Power (MPP)**

The micro pulses within the pulse segments offer the flexibility to stabilize the plasma and control the voltage, current, and power.

Increase High rate deposition
Ionization Zones

The HiPIMS plasma breaks down in isolated ionization zones (IZs) that rotate in the direction given by the ExB drift, with velocities around $10^4$ ms$^{-1}$ and frequencies in the range of about 100 kHz.

- Positive feedback loop between electron mean free path and ionization leads to “bunching” of plasma in ionization zones
- Ionization zones move in ExB direction because ions are “evacuated” from ionization zones by electric field, exposing new neutrals to ionization by drifting electrons
- Electrons drift according to the local E and B fields, perpendicular to both, and produce electron jets related to the azimuthal electric field of the plasma zone
- the physically relevant power density of HIPIIMS is much higher than the typically reported average power density
- Ionization zones explain why HIPIIMS works as observed, and offer “energetic condensation” in the context of sputtering and SRF coatings.

From the target point of view, a HiPIMS discharge represents therefore a situation of continuous temporal and spatial change in local sputtering conditions.

- 3” Nb target, peak current $\sim 200$ A
- reduction of image exposure time gives immediate clues on rotational speed $\rightarrow \sim 10^4$ m/s

Enhanced power density is the key to the desired ionization of sputtered material: films are denser, smoother, and some grain texture control is possible.

Ion Energy Distribution Function


HiPIMS – Applications to SRF Surfaces

**Bias voltage:**
Control ion trajectories & energy in collisionless sheath

**In-situ substrate pre-treatment:**
Cleaning or oxide layer removal – typically with plasma etching (high voltage applied to the substrate)
*Interface engineering* - film forming species are implanted in the substrate, forming a gradient towards the surface
HiPIMS – Applications to SRF Surfaces

- High level of ionization
- High energy ions, tunable with bias voltages
- Film densification
- Film smoothness
- Possibility to control the film structure
- Good surface coverage (even for high aspect ratio objects)
- Enhanced adhesion to substrate
HiPIMS – Applications to SRF Surfaces

Easy extension to other materials
- Relatively straightforward to produce NbN, NbTiN & multilayers
- Possibility to use two different cathode materials with two asymmetrically operating magnetrons to produce Nb$_3$Sn, MgB$_2$, ...

Preliminary TOF study of Nb HIPIIMS with $N_2$ gas: the measured ion species promise the formation of dense, textured NbN

HIPIMS – LBNL

Plasma Studies especially for Nb

HIPIMS with Nb target, \( \phi \, 5 \text{ cm} \)

HIPIMS and Self-Sputtering of Niobium


HIPIMS in 0.25 Pa of Ar

HIPIMS in 0.50 Pa of Kr
Superimpose a Mid Frequency (MF) discharge in between the HiPIMS pulses to lower frequency the HiPIMS pulses

HiPIMS pulses do not depend on MF pulse pattern
As long as the MF discharge is there, the HiPIMS pulses can be spaced as far (or close) in time as desired.

HiPIMS pulses must be longer than 50 μs
Relative power in HiPIMS and MF components is tunable

Nb+ emission lines dominate the emission spectrum starting at moderate voltages
Intensity of all lines increases with increasing discharge voltage
OES peak ratios give some information on changes in relative density of each ionic species

For film growth, applied voltage should be about 650V
discharge right in the low voltage end of Regime III for pulse widths greater than 100 μs
Large population of Nb+ at this voltage, Creation of Nb\(^{2+}\), which doesn’t aid film growth anymore than Nb+, is minimized

Moderate voltage will reduce tendency for arcing
Dedicated Nb-HIPIIMS Chamber

Dual Magnetron: Most effective for a Biasing & influencing Ion Energies and Trajectories

- chamber for 1.3 GHz SRF cavities
- base pressure in the low $10^{-8}$ range
- residual gas analyzer
- 2 small cylindrical, movable magnetrons
- decoupled substrate heating and biasing
- pyrometer 100-600 °C
- 2 SIPP pulsers for dual-HIPIIMS and bias
• dual cylindrical magnetron in at relatively low power sputtering mode
• Dominated by argon emission

• dual cylindrical magnetron in high power mode (above runaway threshold)
• Dominated by niobium emission
HiPIMS – CERN
Collaboration with Sheffield Hallam University

Sheffield Hallam University: Plasma Studies
CERN: 1.3-1.5 GHz cavity deposition

Ion Energy Dispersion Function (IEDF)

G. TERENZIANI, S. CALATRONI, A.P. EHIASARIAN, NB COATINGS FOR SUPERCONDUCTING RF APPLICATIONS BY HIPIMS, 2013-09-11
HiPIMS – CERN

Vertical cylindrical magnetron
Base pressure $10^{-10} – 10^{-11}$ mbar
Movable SmCo magnet

Typical HiPIMS current and voltage behavior

Hüttinger Electronics TruPlasma Highpulse DC Unit:
10 kW max average power delivered
voltage up to 2 kV
pulse width up to 200 us
frequency up to 500 Hz.
HiPIMS – CERN

DC-MS

HIPIMS /150 A peak current

HIPIMS /50 A peak current

HIPIMS /200 A peak current

HIPIMS /150 A peak current

HIPIMS /200 A peak current

HIPIMS /150 A peak current

HIPIMS /200 A peak current
Nb/Cu cavities have been produced both by DC cylindrical magnetron sputtering and cylindrical HiPIMS with Kr

peak current 200 A (2 A/cm²)
ΔV = 570 ± 10 V
Average current 2.6 A
Pulse width 200 µs
frequency 106 Hz.

Note: - Substrate preparation is SUBU (by opposition to electropolishing for the best 1.5GHz DCMS Nb/Cu cavities to date)
- Measurement at higher fields than 11MV/m prevented by interlock system due to radiation
HiPIMS – CERN

Nb/Cu cavities have been produced both by DC cylindrical magnetron sputtering and cylindrical HiPIMS with Kr.

Peak current 200 A (2 A/cm²)
ΔV = 570 ± 10 V
Average current 2.6 A
Pulse width 200 µs
Frequency 106 Hz.

Note: - Substrate preparation is SUBU (by opposition to electropolishing for the best 1.5GHz DCMS Nb/Cu cavities to date)
- Measurement at higher fields than 11MV/m prevented by interlock system due to radiation.
HiPIMS – JLab
Collaboration with LBNL, College William & Mary

Study on samples & cavities

2 coating systems:
- UHV Multi-technique Deposition system
- Cylindrical HiPIMS cavity system

Center stage in development to optimize the sample-target relative positions for Nb and Multilayer films

Bridge studies with energetic condensation
Nb films produced by ECR (Nb\(^+\) ions in UHV)
Niobium cathode
Open grid-like Nb anode
Internal rare earth magnets with field extending entire cathode & cavity length
Penetrating cathode every 1.7 cm producing a peak B field of 0.2 T at a 4 mm distance from the cathode surface with the same longitudinal periodicity.
Water cooled
HiPIMS – JLab

- Ion flux characterization under various pulse conditions
- Ion energy spectrum & Nb ions to neutral ratio. (several anode designs)
- System parameter space & film properties (initially $T_c$ & RRR)

Cavity coating procedure:
- Substrate preparation: CBP + Electropolishing
- Assembly in clean room
- Pump down and bake-out to adequate temperature
- Coating parameters derived from work done with ECR plasma
- Coating with Kr: less trapped in the niobium film, tends to damp plasma instabilities than argon
Conclusions

- HiPIMS is an emerging array of energetic condensation techniques with extensive studies in plasma physics and materials.
- HIPIIMS has the advantage of not generating macroparticles (assuming that arcing is prevented).
- Nb has a relatively low self-sputtering yield → “gasless” self-sputtering in vacuum could not be demonstrated.
- Low pressure operation works well with optimized pulse frequency.
- HIPIIMS cavity systems are ready in the Laboratories involved with HiPIMS.
- Nb/Cu cavities have already been coated at CERN with encouraging results.
- Technique directly applicable to material systems beyond Nb.